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IS 2432 (1993): Liquid sulphur dioxide [CHD 6: Industrial Gases]

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(पहला पुनरीक्षण)

Indian Standard

LIQUID SULPHUR DIOXIDE — SPECIFICATION

(First Revision)

UDC 661.9-498.1 : 546.224-31

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, *9* BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

December ???

Price Group 5

Industrial Gases Sectional Committee, CHD 006

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Industrial Gases Sectional Committee had been approved by the Chemical Division Council.

Liquid sulphur dioxide is used as refrigerant, fumigant, preservative in food industry, bleaching agent, antichlor and as a raw material in the synthetic chemical industry.

In this revision, the method for determination of moisture has been modified, Amendment Number 1 issued to the standard in October 1972 incorporated and the choice of sample containers for liquid sulphur dioxide has been left open to the agreement between the purchaser and the manufacturer.

Wherever a reference to any Indian Standard appears in this specification, it shall be taken at a reference to the latest version of the standard.

In preparing this standard, assistance has been derived from SAA (E) K. 514-1943 Liquid sulphur Dioxide, issued by the Standards Association of Australia and ASTM D 1265-55 Standard Method of Sampling Liquefied Petroleum Gases, issued by the American Society for Testing and Materials.

The composition of the sectional committee responsible for formulating this standard is given in Annex D.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

AMENDMENT NO. 1 FEBRUARY 1998
TO
IS 2432 : 1993 LIQUID SULPHUR DIOXIDE — SPECIFICATION

(First Revision)

(*Second cover page, foreword, second paragraph*) — Delete the words 'preservative in food industry'.

(*Page* 1, *clause* **1**, *line* 3) — Delete the word 'preservative'.

(CHD 6)

Reprography Unit, BIS, New Delhi, India

Indian Standard

LIQUID SULPHUR DIOXIDE — SPECIFICATION

(First Revision)

1 SCOPE

The standard prescribes requirements and methods of sampling and test for liquid sulphur dioxide for use as refrigerant, fumigant, preservative, bleaching agent, antichlor and as a raw material in synthetic chemical.

2 REFERENCES

The Indian Standards listed below arc necessary adjuncts to this standard:

IS No.	*Title*
1070 : 1992	Water for general laboratory use (*third revision*)
1260 (Part 1) : 1973	Pictorial markings for handling and labelling of goods : Part 1 Dangerous goods (*first revision*)
7062 : 1973	Glossary of terms used in gas industry

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 7062 : 1973 shall apply.

4 GRADES

The material shall be of the following two grades:

Grade 1 — for refrigerating and air conditioning industries, and

Grade 2 — for other uses.

5 REQUIREMENTS

5.1 Description

The material shall be colourless, non-flammable liquid possessing a strong suffocating odour characteristic of burning sulphur.

5.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col 5 of Table 1.

6 PACKING AND MARKING

6.1 The material shall be supplied in liquefied condition in cylinders or other containers whose capacity shall be subject to agreement between the purchaser and the supplier.

6.1.1 The cylinders shall comply with the requirements prescribed in the *Gas Cylinder Rules, 1981* of the Government of India, with such modifications as may be ordered from time to time by the Chief Inspector of Explosives, Government of India, or any other duly constituted authority. Other containers, if used, shall also conform to the requirements set out by the above mentioned authority.

6.1.2 The packing, marking and labelling of cylinders shall be in accordance with the requirements for cylinders for liquid gases given in the *Gas Cylinder Rules, 1981* of the Government of India, with such modifications as may

Table 1 Requirements for Liquid Sulphur Dioxide

(*Clause* 5.2)

Sl No.	**Characteristic**	**Requirement for**		**Method of Test (Ref to Cl No. in Annex A)**
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Colour and sediment	To pass test	To pass test	A-2
ii)	Moisture, parts per million (*w/v*), *Max*	50	200	A-3
iii)	Non-volatile residue, parts per million (*w/v*), *Max*	2	1 000	A-4
iv)	Sulphur trioxide and sulphuric acid (as So_3), parts per million (*m/v*), *Max*	10	50	A-5
v)	Non-condensible gases in liquid phase, percent by volume, *Max*	0.02	0.02	A-6

be ordered from time to time by the Chief Inspector of Explosives, Government of India, or any other duly constituted authority. Other containers, if used, shall also be marked and labelled in accordance with the instructions issued from time to time by the above mentioned authority.

6.1.3 The cylinders shall also be marked with an appropriate symbol in accordance with IS 1260 (Part 1) : 1973.

7 SAMPLING

Representative samples of the material shall be drawn as prescribed in Annex B.

ANNEX A

(*Clause* 5.2)

ANALYSIS OF LIQUID SULPHUR DIOXIDE

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwse, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 TEST FOR COLOUR SEDIMENT

A-2.1 Procedure

Take 250 ml of the material in a flask which is immersed in methyl alcohol to prevent the formation of ice on its outside surface. Compare it with the same volume of water contained in a similar flask in diffused daylight.

A-2.1.1 The material shall be considered to have passed the test if its colour is not darker than that of water and also if no sediment or suspended matter is visible.

A-3 DETERMINATION OF MOISTURE AND WATER VAPOUR

A-3.0 General

Two methods have been prescribed, namely, Karl-Fischer and, frost or dew point hygrometer method. However, in case of dispute, Karl-Fischer method will be the referee method.

A-3.1 Karl-Fischer Method

A-3.1.0 *General*

The reagents used in the method are sensitive to water and precautions shall be taken throughout to prevent exposure to atmospheric moisture.

A-3.1.1 *Apparatus*

A-3.1.1.1 *Titration flask*

With a capacity of 60 to 75 ml, fitted with a rubber stopper with accommodation for the burette tip and a vent tube provided with a desiccant.

A-3.1.1.2 *Stirrer* — magnetic or propeller type.

A-3.1.2 *Reagents*

A-3.1.2.1 *Methanol* — dried over burnt quick lime.

A-3.1.2.2 *Pyridine* — dried over sodium hydroxide pellets.

A-3.1.2.3 *Karl Fischer reagent*

Dissolve 63 g of iodine in 100 ml of pyridine. Cool in ice and pass sulphur dioxide gas into the solution until a gain in weight of 32.3 g has occurred, taking care to avoid absorption of atmospheric moisture. Add sufficient methanol to produce 500 ml. Allow to stand for 24 hours.

A-3.1.2.4 *Standardization of Karl Fischer reagent*

Add about 20 ml of methanol to the titration vessel and titrate to the end point with Karl Fischer reagent without recording the volume required. Introduce by means of Lunge's pipette a suitable amount of water, accurately weighed, and titrate again with Karl Fischer reagent. The end point can be detected visually by the change from a light brownish-yellow to an amber colour. Calculate the water equivalent of the reagent in milligrams of water per millilitre.

NOTE — Karl Fischer reagent deteriorates continuously and should be standardized immediately before use or daily, as required. When freshly prepared, one millilitre is equivalent to about 5 mg of water.

A-3.1.3 *Procedure*

Transfer into the titration vessel an accurately measured volume of liquid sulphur dioxide. Titrate with Karl Fischer reagent to the end point, the liquid being stirred efficiently during titration.

A-3.1.4 *Calculation*

$$\text{Moisture, parts per million (m/v)} = 1000 \times \frac{A V_1}{V_2}$$

where

A = water equivalent of Karl Fischer reagent in mg/ml as obtained in **A-3.1.2.4**,

V_1 = volume in ml of Karl Fischer reagent used in the titration in **A-3.1.3**, and

V_2 = 5 volume in ml of liquid sulphur dioxide taken for the test.

A-3.2 Frost or Dew Point Hygrometer Method

A metal surface on the hygrometer is cooled so that dew or frost is formed from the moisture content of the gas at a particular pressure which may be observed optically in the apparatus. The temperature at which the dew or frost is formed is a measure of moisture content of the gas. The procedure to be followed for determining the moisture content shall depend upon the type of the apparatus to be used. The manufacturer's instructions in this regard shall be followed (*see* Annex C).

A-4 DETERMINATION OF NON-VOLATILE RESIDUE

A-4.1 Procedure

A-4.1.1 Take a 400-ml conical flask with a calibration mark at 250-ml point. Thoroughly clean the flask with chromic acid cleaning solution and water. The final water rinsing shall be neutral to methyl red indicator. Dry the flask at about 120°C. The surface of the flask shall be bright and free from film. Weigh the flask accurately.

A-4.1.2 Wipe the valve of the sample container containing the sample, flush it and draw 250 ml of the material in the flask. Stopper the flask with a plug of cotton wool and allow the material to evaporate completely. Again take in another volume of 250 ml of the material and allow to evaporate.

A-4.1.3 Remove the last traces of sulphur dioxide gas by connecting a gooch crucible adapter to suction and applying the rubber end to the flask, breaking contact after a short time. Repeat suction until no odour of sulphur dioxide remains. Weigh the flask again. Preserve the flask containing the non-volatile residue for test in **A-5.**

A-4.2 Calculation

$$\text{Non-volatile residue, parts per million (} m/v \text{)} = 2 (M_2 - M_1) \times 10^3$$

where

M_2 = mass in g of the flask after evaporation, and

M_1 = mass in g of the empty flask.

A-5 DETERMINATION OF SULPHUR TRIOXIDE AND SULPHURIC ACID

A-5.1 Reagents

A-5.1.1 *Standard Sodium Hydroxide Solution* — 0.01 N.

A-5.1.2 *Phenolphthalein Indicator Solution*

Dissolve 0.1 g of phenolphthalein in 60 ml of rectified spirit (*see* IS 323 : 1959) and dilute with water to 100 ml.

A-5.2 Procedure

Add 200 ml of water to the flask containing non-volatile residue (*see* **A-4.1.3**). Shake it well and titrate with standard sodium hydroxide solution using phenolphthalein as indicator.

A-5.3 Calculation

$$\text{Sulphur trioxide and sulphuric acid (as } SO_3 \text{), parts per million (} m/v \text{)} = 80\, VN$$

where

V = volume in ml of standard sodium hydroxide solution used, and

N = normality of standard sodium hydroxide solution.

A-6 DETERMINATION OF NON-CONDENSIBLE GASES IN LIQUID PHASE

A-6.1 Apparatus

The assembly of apparatus is shown in Fig. 1.

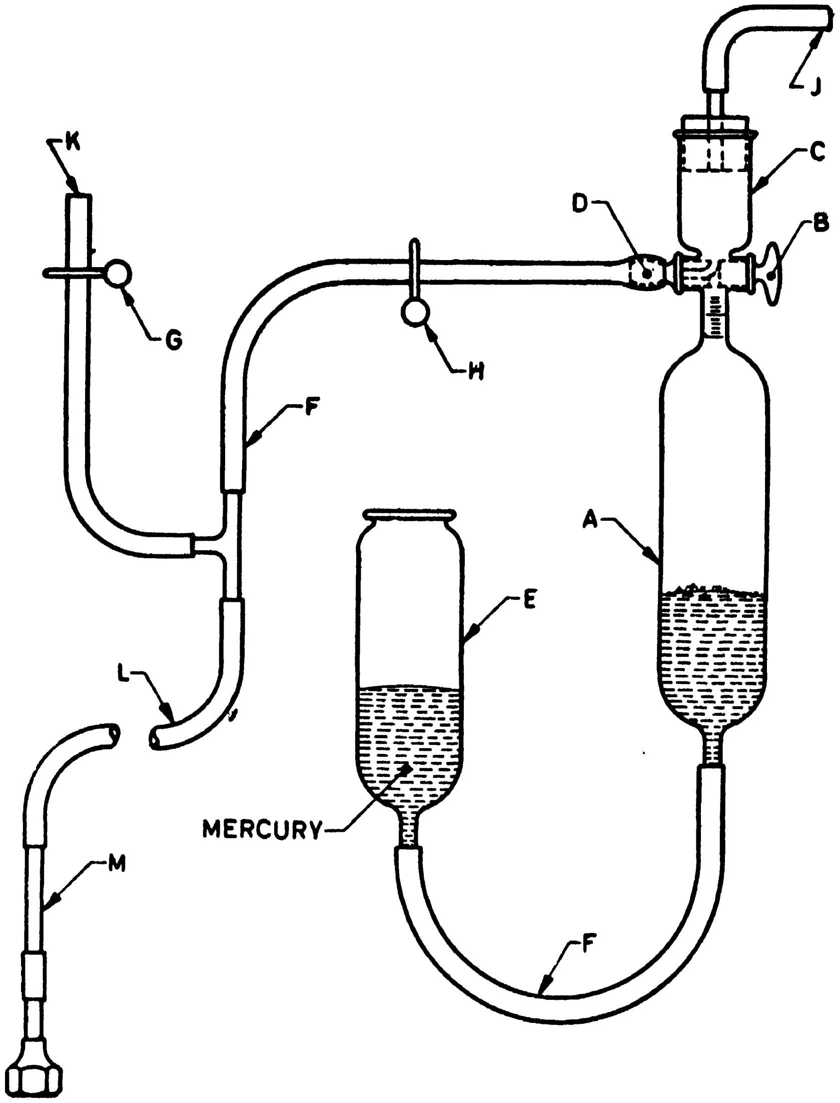
A-6.2 Reagent

A-6.2.1 *Potassium Hydroxide Solution* — approximately 30 percent.

A-6.3 Procedure

A-6.3.1 Open the tail type stopcock *B* so as to connect *A* and C, keeping the pinch-cock *H* closed. Raise the levelling bulb *E* until mercury just enters the cup *C*. Close cock *B* leaving a small globule of mercury in the cup *C*.

A-6.3.2 Place the sample container containing the material on its side so that the remshorn of the lower valve is in the liquid phase, and connect the apparatus to the sample container as shown in Fig. 1.



A – 100 ml gas burette, with the upper tip graduated in 0.02 ml down to 0.2 ml mark
B — Three way tail type stopcock
C — Cup for addition of potassium hydroxide
D – Connection for sulphur dioxide inlet
E – Levelling bulb (containing mercury)
F — Rubber tube
G, H — Pinchcock
J, K — Open ends of rubber tubing
L — Rubber tubing from sample container
M — Glass sight tube near point of attachment to sample container

FIG. 1 APPARATUS FOR DETERMINATION OF NON-CONDENSIBLE GASES IN LIQUID PHASE

A-6.3.3 Keeping the pinch-cock *G* open, cautiously open the sample container valve until liquid sulphur dioxide is seen to run through the glass tube *M* and overflow at *K*. Open the pinch-cock *H* and turn the stopcock *B* to position shown in the figure after which the pinch-cock *G* and the sample container valve shall be closed.

A-6.3.4 When the liquid sulphur dioxide has vaporized as indicated by the globule of mercury, open the sample container valve for two or three minutes to sweep out the air in the apparatus. Keeping the pinch-cock *G* closed, turn the stopcock *B* quickly to connect the sample container with burette *A* with sulphur dioxide gas. Close the pinch-cock *H,* open the pinch-cock *G* and turn stopcock *B* so as to connect *A* and C allowing the burette full of gas to run to waste from the cup *C.*

A-6.3.5 Refill the burette *A* in the same manner as indicated in **A-6.3.1** with exactly 100 ml of sulphur dioxide gas under atmosphere pressure. Close pinch-cock *H,* turn the stopcock to position shown in the figure. Add 15 to 20 ml of potassium hydroxide solution in the cup *C* and introduce it in the burette through the stopcock taking care to exclude all air. After contraction has ceased, adjust the level of mercury in the bulb to that in the burette and read the residual volume.

A-6.4 Report the volume in millilitre of the residual gas as percent of non-condensible gases in liquid phase.

ANNEX B

(*Clause* 7)

SAMPLING OF LIQUID SULPHUR DIOXIDE

B-1 GENERAL

B-1.1 Samples shall be drawn and stored in a protected but well ventilated place.

B-1.2 Precautions shall be taken to protect the sample, the material being sampled, the sampling apparatus and the containers for samples from adventitious contamination,

B-1.3 The sample containers shall be of two-valve type as shown in Fig. 2 or as agreed to between the purchaser and the manufacturer and shall comply with the requirements prescribed for cylinders in **6.1.1.**

B-1.4 The sample containers shall be filled with the material in liquid phase up to not more than 80 percent of their capacity.

B-1.5 Each sample container shall be marked with all the details marked on the container of material (*see* **6.1.2**) and the date of sampling.

B-1.6 Samples shall be drawn under the supervision of persons familiar with the necessary safety precautions.

B-1.7 Samples shall be taken from liquid phase only.

B-1.8 The material shall be mixed as thoroughly as possible by suitable means, and then sample drawn from any point of the container. If it is not possible to mix the material until homogeneity is assured, a liquid sample shall be secured as agreed to between the purchaser and the supplier.

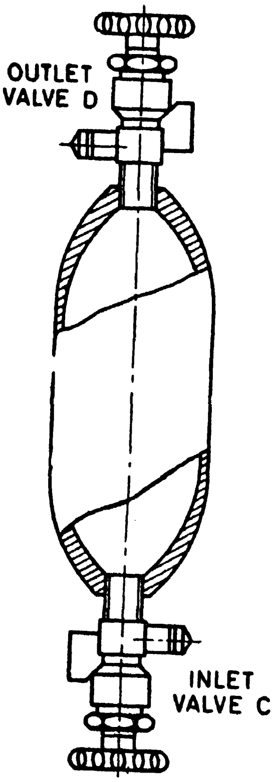


FIG. 2 Two-VALVE SAMPLE CONTAINER

B-2 SCALE OF SAMPLING

B-2.1 Lot

B-2.1.1 If material is supplied in tanks (1 000 kg or above), each tank shall constitute a lot.

B-2.1.2 In case of cylinders, a lot shall consist of all cylinders charged during one work shift from one charging manifold.

B-2.2 For ascertaining the conformity of the material to the requirements of the specification, samples shall be tested separately for each lot.

B-2.2.1 In the case of cylinders, the number of cylinders to be selected from lots of different sizes shall be in accordance with Table 2.

B-2.3 The cylinders to be selected for sampling shall be chosen at random. In order to ensure randomness, arrange all cylinders in a systematic manner and starting from any one, count them as 1, 2, 3, etc, up to *r* and so on, where *r* is the integral part of *N/n*. Every *r*th container thus counted shall be withdrawn to constitute the sample.

Table 2 Number of Containers to be Selected for Sampling

(*Clauses* B-2.2.1 *and* B-2.3)

Lot Size (1) *N*	Sample Size (2) *n*
2 to 15	2
16 to 40	3
41 to 75	4
76 to 125	5
Above 125	6

B-2.4 From each cylinder selected for sampling, a small portion of the material, in liquid phase, sufficient for carrying the tests in duplicate, specified in Annex A shall be drawn (*see* **B-3**). These samples shall constitute the individual test samples.

B-3 PROCEDURE

B-3.1 Sample Transfer Line

A sample transfer line made of materials on which sulphur dioxide has no action shall be used. A satisfactory line is one equipped with two valves on the sample container end, a control valve *A* and a vent valve 5, as shown in Fig. 3.

B-3.2 Purging Sample Transfer Line

Connect the ends of the transfer line securely to the product source and to the inlet valve *C* of the sample container as shown in Fig. 3. Close the control valve *A*, the vent valve *B*, and the inlet valve *C*. Open the valve at the product source and purge the transfer line by opening the control valve *A* and the vent valve 5, till liquid escapes through vent valve *B*.

B-3.3 Purging Sample Container

With the sample container in an upright position and its outlet valve *D* at the top, close vent valve *B*, and inlet valve *C* and open control valve *A*. Open inlet valve *C*, and partly fill the container with sample by slowly opening the outlet valve *D*. Close the control valve *A*, and allow part of the sample to escape in the vapour phase through outlet valve *D*. Close outlet valve *D* and release the remainder of the sample in the liquid phase by opening vent valve *B*. Repeat the purging operation at least three times.

B-3.4 Transfer of Sample

Close the vent valve *B*, open the control valve *A* and inlet valve *C*, and fill the container with sample. Close the inlet valve *C*, and the valve at the product source. Open the vent valve *B*. After the pressure is fully released.

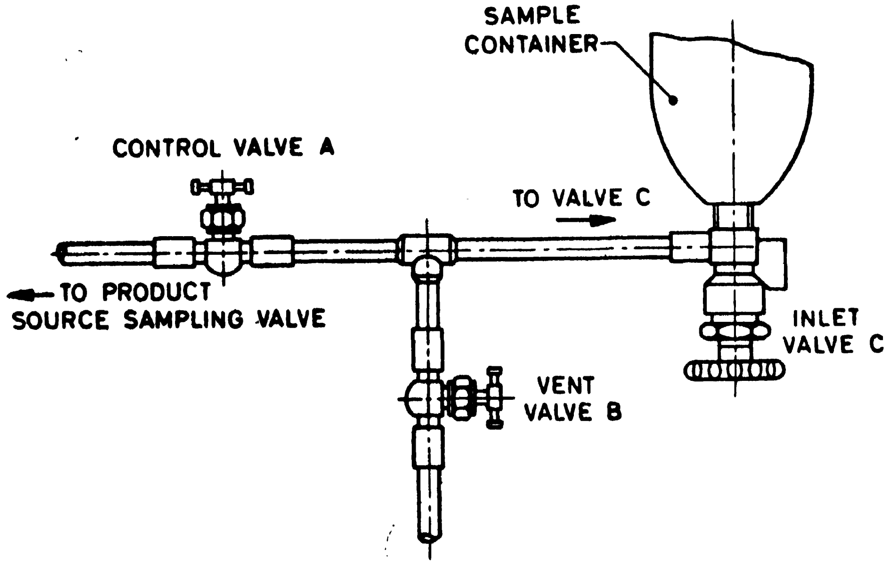


FIG. 3 SAMPLE TRANSFER LINE

disconnect the transfer line from the product source and from the sample container. Immediately after obtaining the sample, provide a 20 percent ullage in the sample container.

B-3.5 Care of Samples

Place samples in a cool place as soon as possible. Keep them there until all tests have been completed. Discard samples in any sample container which develops leaks. Protect the valves of the sample container, either by packing the container in a crate or by using a protective cap, so that accidental damaging of the valve or tampering with it is avoided.

B-4 NUMBER OF TESTS

B-4.1 Tests for all the characteristics given in Table 1 shall be performed on each of the individual samples.

B-5 CRITERION FOR CONFORMITY

B-5.1 For Colour and Sediment

The lot shall be taken as having conformed to the prescribed requirement if each sample passes the test.

B-5.2 For characteristics other than colour and sediment, calculate the mean ($\bar{X}$) and range (R) of the test results (range being defined as the difference between the maximum and the minimum values of test results).

B-5.2.1 The lot shall be declared as conforming to the prescribed requirements for the different characteristics if the corresponding value computed for ($\bar{X}$ + 0 6 R) does not exceed the prescribed requirements.

ANNEX C

(*Clause* A-3.2)

HYGROMETRIC UNIT EQUIVALENCE TABLE

$$\text{ppm}\ (W) = \text{ppm}\ (V) \times \frac{\text{Molecular weight of water}}{\text{Molecular weight of carrier gas}}$$

Dew Point (1)	ppm *(V)* at 1 kg/cm^2 (2)	ppm *(W)* SO$_2$ (3)	Dew Point (1)	ppm *(V)* at 1 kg/cm^2 (2)	ppm *(W)* SO$_2$ (3)
—80	0.54	0.151 7	—38	158.73	44.594 0
—78	0.75	0.210 7	—36	197.91	55.601 4
—76	1.03	0.289 4	—34	245.86	69.072 6
—74	1.41	0.396 1	—32	304.34	85.502 1
—72	1.91	0.536 6	—30	375.40	105.485 6
—70	2.58	0.724 8	—28	461.49	129.652 3
—68	3 47	0.974 9	—26	565.44	158.856 3
—66	4.63	1.300 8	—24	690.55	194.005 0
—64	6.15	1.727 8	—22	840.68	236.182 9
—62	8.13	2.284 1	—20	1 020.29	286.643 0
—60	10.68	3.000 5	—18	1 234.56	346.840 6
—58	13.96	3.922 0	—16	1 489.45	418.450 1
—56	18.16	5.101 9	—14	1 791.84	503.404 4
—54	23.52	6.607 8	—12	2 149.61	603.917 3
—52	30.31	8.515 4	—10	2 571.83	722.536 9
—50	38.89	10.925 9	— 8	3 068.86	862.173 9
—48	40.67	13.954 4			
—46	63.18	17.750 0	— 6	3 652.55	1026.157 3
—44	80 02	22.481 0	— 4	4 336.39	1 218.277 2
—42	100.94	28.358 4	— 2	5 135.78	1 442.860 0
—40	126.82	35.629 2	0	6 068.81	1 704.988 0

ANNEX D

(Foreword)

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DR R. K. SINGH Director (Chem)	Director General, BIS (*Ex-officio Member*)

Member Secretary

SHRI R. NARULA
Joint Director (Chem), BIS

(*Continued on page* 9)

(*Continued from page* 8)

Sulphur Dioxide, Ammonia, Chlorofluoro Hydrocarbon and Other Refrigerants Subcommittee, CHD 006 : 02

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